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TECHNICAL REPORT
C&OM-13

RESEARCH COMPOUNDING OF NITROSO RUBBER

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by
Charles B. Griffis

Clothing and Organic Materials Division

OCTOBER 1965

U. S. ARMY NATICK LABORATORIES
Natick Massachusetts

US ARMY MATERIEL COMMAND

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1C024401A329

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FOREWORD

Presently available commercial rubbers do not meet all of the needs of military equipment. Special rubber compounds are required in many items to assure their global operational capabilities. This is particularly true of those items containing rubber that must be in contact with gasoline, such as lightweight gasoline hose that can be reeled at -65°F, flexible gasoline containers that have a 10,000-gallon capacity and are easy to handle, and lightweight insulated boots that are soft and flexible at -65°F, all of which help to improve the efficiency not only of the military man but also of his equipment.

The Army is continuing to conduct research on the development of new kinds of rubber that can be compounded to increase its military capability. Nitroso rubber is one such product. Nitroso rubber was first produced in the laboratories of the Minnesota Mining and Manufacturing Company under Government contract. It has now been made in pilot-plant quantity by the Thiokol Chemical Corporation under U. S. Army Natick Laboratories Contract No. DA19-129-AMC-69. Research compounding has been performed at the U. S. Army Natick Laboratories to develop information that the rubber technologist can use for detail compounding for specific end-item application.

S. J. KENNEDY
Director
Clothing & Organic Materials Division

Approved:

DALE H. SIELING, Ph.D.
Scientific Director

W. W. VAUGHAN
Brigadier General, USA
Commanding

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ABSTRACT

Nitroso rubber, a copolymer of trifluoronitrosomethane and tetrafluoroethylene, has been made in pilot-plant quantity by the Thiokol Chemical Corporation under Army Contract DA19-129-AMC-69. Research compounding studies have been made on this pilot-plant production rubber. A variety of cross-linking (vulcanization) agents were investigated, the most effective of which was triethylenetetramine. Of a series of reinforcing fillers used, the best physical properties were obtained with HiSil 303. However, a silicone-treated HiSil 233 gave comparable results.

The curing characteristics of the nitroso rubber made in the pilot plant at the Thiokol Chemical Corporation were different from those of the material prepared in the laboratory of the Minnesota Mining and Manufacturing Company. Based on the research compounding studies made, there is no indication that rubber compounds having excellent physical properties can be made from the Thiokol Chemical Corporation nitroso rubber.

RESEARCH COMPOUNDING OF NITROSO RUBBER

1. Introduction

Rubber research and development is of vital importance for the success of military operations under all climatic conditions. Presently available rubbers limit the operational capabilities of many military items under extreme temperature conditions. Nitroso rubber, a new copolymer that has emerged from the research and development program, has potentiality for improving the temperature dependence of many of these items.

Nitroso rubber is a copolymer of trifluoronitrosomethane and tetrafluoroethylene that was developed under Contract DA19-129-QM-1684 with the Minnesota Mining and Manufacturing Company. This work[1,2,3] established the basic properties of the material and indicated that only amines will cross-link it.

The physical properties obtained from amine vulcanized (cross-linked) nitroso rubber were poor as compared to many other synthetic rubbers. It was believed that a more complete compounding study than had previously been possible because of the scarcity of the nitroso rubber would improve these properties. Therefore, work was initiated, under Contract No. DA19-129-AMC-69, for the Thiokol Chemical Corporation to develop techniques for producing this rubber in pilot plant quantities. By means of these techniques, approximately 200 pounds of rubber was produced. This report is concerned with the research compounding of this rubber.

2. Materials and Test Methods

The Thiokol Chemical Corporation produced six batches of nitroso rubber that were used in this study. These rubbers, all copolymers of trifluoronitrosomethane and tetrafluoroethylene, were made in the pilot plant of the company.

Compounding studies have been conducted on only two of the batches.

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1. Minnesota Mining and Manufacturing Company, Arctic Rubber. Research Rept. (Contract DA19-129-QM-1684) 23 Dec 1962

2. Montermoso, J.C., C.B. Griffis, A. Wilson and G.H. Crawford. Vulcanization and Properties of Nitroso Rubber. Rubber and Plastics Age, 42 (5) May 1961

3. Griffis, C.B. and M.C. Henry. Nitroso Rubbers. Rubber and Plastics Age, 46 (1) Jan 1965

Their physical properties and the test methods used are as follows:

Property	ASTM Test Method Number
Tensile strength Ultimate elongation Stress at 300% elongation	D412-62T
Hardness	D676-59T
Mooney viscosity	D1646-63

The differential thermal analysis data were obtained with a Du Pont Model 900 differential thermal analyzer under the following conditions:

Sample size:	4mm	Atmosphere:	Ar_2 @ 760mm
Reference:	glass beads	Temperature scale:	50
Program mode:	heat	Δ Temperature scale:	0.5
Rate of heating:	10^{o}/min	Base line slope:	0

3. Results and Discussion

It was immediately noted that the properties of the vulcanizates produced from the new lot of nitroso rubber (Thiokol) were not similar to those of the compounds from the earlier rubber (Minnesota Mining and Mfg. Co.) even though the compounding recipe used was the same. For example, while tensile strengths of 1000 psi or more had readily been obtained from the earlier rubber (MMM), tensile strengths of 300 psi represented the maximum obtainable from the Thiokol rubber. There were differences in curing characteristics (scorch time, time of cure, and cure index) as well. To compensate for these differences, a series of amine cure studies was made.

The compounding and curing recipes, cure times and temperatures, and subsequent physical properties of various compounds of batch 5702 are given in Tables I and II, and of batches 5702 and 5675 in Tables III and IV. Table I gives the test results of amine-cured, HiSil 303-filled vulcanizates and Table II the results with amine-cured, carbon black-filled vulcanizates. Table III lists the vulcanizates with amine cures and amine and filler variations, and Table IV those with other than amine cures.

In the Table I series (HiSil 303 filler), the triethylenetetramine (TETA) was varied from 1.25 to 5 pphr (parts per hundred rubber) and the hexamethylenediamine carbamate (Diak #1) from 1 to 2.5 pphr. Press cure temperatures varied from 220^{o} to 260^{o}F. The Thermax black filler which had been found to increase the rate of cure of the Thiokol rubber, was tried in combination with the HiSil 303 in compound 57. This vulcanizate proved to be too weak to test. Compound 79, which had the smallest amount of TETA (1.75 pphr), was the best of this series but even this showed a tensile strength of only 245 psi.

In the second series using the amine cure (Table II), various carbon black fillers were used: medium thermal (MT) furnace black "Thermax", an easy-processing channel (EPC) black, and a high-abrasion furnace (HAF) black. Press cure temperatures varied from 180° to 250°F. None of the black fillers gave vulcanizates that were superior to those using HiSil 303 (Table I) or to those using Linde Silicone treated HiSil 233 or Silstone 101. When the curing time was kept below 250°F, only the HAF black among the black-filled compounds produced sponging. At 210°F, the HAF black compound did not sponge but it failed to cure.

Table III describes the compounds made with a variety of amine cross-linking agents, fillers, and stabilizers. It had been found that the addition of more than 2 pphr of triethylenetetramine usually produces sponging and always lowers the tensile strength. Sponging often is not readily discernible; sometimes it can be detected only by means of a microscope. To insure against sponging, compound 14 was made using only 1 pphr of triethylenetetramine and 0.33 pphr of hexamethylenediamine carbamate. To achieve maximum cure with this low level of curative, the compound was press-cured for 240 minutes at 210°F and then oven-cured for 24 hours at 195°F. The tensile strength of this compound (300 psi) was the highest obtained with the Thiokol nitroso rubber. Compound 14 showed no signs of sponging. None of the other variations produced a vulcanizate with more than 200 psi tensile strength.

Table IV shows the results when compounds were vulcanized with other than amine cross-linking agents. None of these vulcanizates could be tested; from visual examination none appeared to have been cured.

To determine the curing characteristics of the nitroso rubber, the viscosity, not only of batches 5702 and 5675 but also of the remaining four batches, was determined on the Mooney viscometer (Table V). Batch 5675 gave the lowest result, a viscosity of only 22. The curing characteristics of a variety of compounds are given in Table VI. Compounds 12 and 13, which represent MMM and Thiokol nitroso rubbers, respectively, showed great differences in curing characteristics. Compound 7 is a compound in which 1 pphr of 1,4 cyclohexane bis (methylamine) replaced 1 pphr triethylenetetramine and then 3 pphr of zinc oxide stabilizer was added. This compound gave the best curing characteristics, hence the same formula was used in a series of compounds (8,9,11, and 12) in which the fillers were varied by the use of different carbon blacks and the effect of zinc fluoride was investigated.

With Thermax black as a filler (compound 9), the zinc fluoride improved the curing characteristics of the nitroso rubber, as is shown below:

No.	Compound	Time for Cure (beyond 35 min) (min)
8.	No filler, no zinc fluoride	no cure
9.	Thermax black with zinc fluoride	5.5
12.	No filler with zinc fluoride	no cure
11.	Thermax black, no zinc fluoride	14.5

Zinc fluoride in HAF black filler systems (compounds 13, 14, and 15) did not change the curing characteristics of these compounds to the same degree that it did with the Thermax black and it did not improve the physical properties of the vulcanizates. In compounds 38 and 41, 2.5 pphr of triethylenetetramine and mixtures of carbon black and silicone-treated fillers were used and the temperature was reduced to 210°F (to eliminate sponging). The cure characteristics produced were excellent but the physical properties were poor.

Results of attempts to identify cross-linking of the nitroso rubber by the use of differential thermal analysis techniques are given in Table VII. Triethylenetetramine was the only material tested that indicated cross-linking, and this occurred at 60°C. Using this method of analysis, the second order transition of -50°C compares with that previously reported[1] on the MMM rubber. Table VII shows that the addition of triethylenetetramine reduced the temperature for the onset of deterioration from 220°C to 200°C. The addition of Diak #1 (hexamethylenediamine carbamate) significantly reduced the temperature for the onset of deterioration. This was also true when other amines were used, and when dicumylperoxide was added.

4. Conclusions

The Thiokol Chemical Corporation nitroso rubber has different curing characteristics from that made by the Minnesota Mining and Manufacturing Company.

The physical properties of the compounds made and reported here, using the Thiokol Chemical Corporation nitroso rubber, were extremely poor.

The reinforcing fillers HiSil 303 and the silicone-treated HiSil 233 were superior to the medium thermal furnace blacks, the high-abrasion furnace blacks, and the easy-processing channel blacks.

5. Recommendation

It is recommended that no development compounding be performed with the Thiokol Chemical Corporation nitroso rubber.

1. Minnesota Mining and Manufacturing Co., op. cit.

TABLE I

COMPOUNDING RECIPES AND TEST RESULTS
AMINE CURES - HiSIL 303 FILLER

Ingredient Parts by Weight	Compound Number 79	50	51	51	57	58	60	64	65
Nitroso, Thiokol 5702	100	100	100	100	100	100	100	100	100
Triethylenetetramine	1.25	2	3	3	3	1.5	5	1.5	1.5
Hexamethylenediamine carbamate	2.5	2	1.25	1.25	1	2	1	1	
HiSil 303	15	20	15	15	15	15	15	7.5	10
Mg O					5				
Zn silicate fluoride					3				
MT black					15				
Sodium borate								7.5	
Press cure (time min/ temp °F)	60/250	60/220	60/260	60/230	60/220	60/260	60/220	60/220	60/220
Oven cure (time hr/ temp °F)	16/212	16/212				64/212			
Tensile strength,(psi)	245	200	sponged	sponged	too weak to test	100	175	too weak to test	too weak to test
Ultimate elongation(%)	380	100				300	260		
Stress at 300% elong. (psi)	230	-				100	-		
Hardness, Shore A	57	80				60	67		

TABLE II

COMPOUNDING RECIPES AND TEST RESULTS
AMINE CURES - CARBON BLACK FILLERS

Ingredient Parts by Weight	Compound Number 9*	10	11	13	17	18	19	22	23	31	32
Nitroso, Thiokol 5702	100	100	100	100	100	100	100	100	100	100	100
Silstone 101										20	20
Triethylenetetramine	3	3	3	3	1.5	1.5		2	3	2.5	2.5
Hexamethylenediamine carbamate	1	1	1	1	1	1	2		1	1	1
1,4 Cyclohexane bis(methylamine)	1	1	1	1	1	1	1	1	1		
ZnO	3	3	3	3	3	3		3	3	3	3
Zinc fluoride	1	1		1							
HAF black				15	25	17	17	17	17		
MT black	15	15	15			8	8	8	8		
Linde silicone-treated Hi Sil									10		
Press cure (time min/temp °F)	60/250	60/210	60/220	60/200	60/200	60/200	60/200	60/200	60/200	60/240	60/210
Oven cure (time min/temp °F)	16/212							16/200	16/200		
Tensile strength (psi)	125	100	140	100	210	200	160	90	200	250	220
Ultimate elongation(%)	290	400	490	370	100	160	225	490	350	750	650
Stress @ 300% elong. (psi)	-	90	100	-	-	-	-	-	-	100	100
Hardness, Shore A	36	30	28	40	62	50	35	38	50	37	41

*Sponged

TABLE II (cont'd)
COMPOUNDING RECIPES AND TEST RESULTS
AMINE CURES - CARBON BLACK FILLERS

Ingredient Parts by Weight	Compound Number 33	34	35	36	37	38	39	40	41	42
Nitroso, Thiokol 5702	100	100	100	100	100	100	100	100	100	100
Silstone 101	20	10	10	10	10	10	10	10	10	10
Triethylenetetramine	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Hexamethylenediamine carbamate	1	1	1	1	1	1	1	1	1	1
ZnO	3	3	3	3	3	3	3	3	3	3
MT black		10	10	10						
EPC black					10	10	10			
HAF black								10	10	10
Press cure (time min/temp °F)	60/180	60/240	60/210	60/180	60/240	60/210	60/180	60/240	60/210	60/180
Tensile strength (psi)	50	150	200	100	100	150	150	sponged	too weak to test	too weak to test
Ultimate elongation (%)	950	500	600	950	500	550	600			
Hardness, shore A	33	26	26	22	28	32	29			

TABLE III

COMPOUNDING RECIPES AND TEST RESULTS
AMINE CURES - AMINE AND FILLER VARIATIONS

Ingredient Parts by Weight	Compound Number 1	2	3	14	4	5	6	7	15	8	21	24
Nitroso, Thiokol 5675	100	100	100	100								
Nitroso, Thiokol 5702					100	100	100	100	100	100	100	100
ZnO	3					5				3	3	
Triethylenetetramine	2	3	3	1	3			2	3	2	1	3
Hexamethylenediamine carbamate			2	0.33	1	2		2	3	1	1	1
Diethylene glycol			1									
1,4 Cyclohexane bis (methylamine)							5	2	3	3	1	
HiSil 233	15		15									
Cab-O-Sil		15										
Silstone 101				10	15	15	15	10	12.5	12.5		15
DyPhos						10						
Nitroso-treated HiSil 233											12.5	
Press cure (time min/temp °F)	60/240	90/210	60/210	240/212	60/190	60/280	60/280	60/280	60/230	60/250	60/200	60/200
Oven cure (time hrs/temp °F)	16/212	16/212	16/212	24/195	16/190	16/300	16/212	-	-	16/212	16/200	16/212
Tensile strength (psi)	sponged	sponged	sponged	300	sponged	no cure	100	sponged	no cure	200	100	180
Ultimate elongation (%)				500			170			700	950	340
Stress at 300% elong (psi)				150			-			-	-	-
Hardness, Shore A				34			60			16	21	53

TABLE III (cont'd)

COMPOUNDING RECIPES AND TEST RESULTS
AMINE CURES - AMINE AND FILLER VARIATIONS

Ingredient Parts by Weight	Compound Number 70	71	72	73	74	75	76	77	78	79	88	81
Nitroso, Thiokol 5702	100	100	100	100	100	100	100	100	100	100	100	100
Triethylenetetramine	2	4	8	1	2					1	5	2
Hexamethylenediamine carbamate	2	5	5			3			2		1	
Ethylenediamine carbamate								3				
Sulfur					1.5	2	2	2	2	1		0.5
Pyrometallicanhydride				5								
Methylene bis (phenyldi-isocyanate)				1								
Sodium bicarbonate				3								
DyPhos	5	5	5									
HiSil 303				15	15	20	20	20	20	20	20	
N,N'-Dicinnamylidene-1,6 hexanediamine							3					
ZnO										3		3
Cadmium stearate										1.5		
Diphenylnitrosoamine											2	
HiSil 233												15
Diethylene glycol												2
Press cure (time min/temp ^{o}F)	60/220	60/230	60/230	180/212	30/250	30/250	30/250	30/250	30/250	30/250	60/250	60/240
Oven cure (time hrs/temp ^{o}F)	-	-	-	-	-	-	-	-	-	1	1	16/212
Tensile strength (psi)	100	100	150	no cure	no cure	no cure	no cure	no cure	150	no cure	no cure	no cure
Ultimate elongation (%)	900	400	225						395			
Stress at 300% elong (psi)	-	-	-						-			
Hardness, Shore A	26	32	42						60			

TABLE IV
COMPOUNDING RECIPES AND TEST RESULTS
OTHER THAN AMINE CURES

Ingredient Parts by Weight	Compound Number 82	83	84	85	86	87	88
Nitroso, Thiokol 5675	100						
Nitroso, Thiokol 5702		100	100	100	100	100	100
Chromiumtrifluoroacetate	5						
Ca O	2						
Cab-O-Sil		15					
Hexamethylenediamine carbamate		1					
Diethylthiourea		2					
MAPO		5					
HiSil 303			15	15	15	15	15
$Bu_2Sn\ Cl_2$			3				
DiCup 40c				9			
Trimethylolpropane trimethacrylate				3			
Pyrometalliccanhydride					1		
MDI					1	1	
MgO							5
Cadox BSG paste							3
Press cure (time min/temp °F)	60/240	60/250	60/300	60/300	60/250	60/250	60/250
Tensile strength (psi)	no cure	no cure	no cure	no cure	no cure	no cure	no cure
Ultimate elongation (%)							
Stress @ 300% elong (psi)							
Hardness, Shore A							

TABLE V

MOONEY VISCOSITY OF VARIOUS
BATCHES OF NITROSO RUBBER

Thiokol Chemical Corporation Batch Identification	Mooney Viscosity (ML-4+1 @ 212 °F)
5702	35
5812	35
5812 (9/14/64)	36
5812 (9/15/64)	35
5812 (Part #3)	36
5675	22

TABLE VI

CURE CHARACTERISTICS AS DETERMINED WITH MOONEY VISCOMETER (LARGE ROTOR)

Ingredient Parts by Weight	Compound Number 12	13	7	8	9	11	12	13	14	15
Nitroso, MMM 9690		100								
Triethylenetetramine	3	3	2	2	3	3	3	3	3	3
Hexamethylenediamine carbamate	1	1	1	1	1	1	1	1	1	1
Silicone-treated HiSil 233	10	10	12.5							
Nitroso, Thiokol 5702	100		100	100	100	100	100	100	100	100
Zinc fluoride					1		1	1		1
Thermax					15	15				
ZnO			3	3	3	3	3	3	3	3
1,4,Cyclohexane bis(methylamine)			1	3	1	1	1	1	1	1
HAF black								15	15	12.5
Temp. of test (°F)	215	215	250	250	250	250	250	250	250	250
Minimum viscosity	11.5	70	56	13	20	8	9	68	54	55
Scorch time (t_5 min)	50	10	5	14.5	2.75	4.2	11.5	7	7.2	4.3
Time of cure (t_{35} min)	100	26.5	18	-	5.5	-	14.5	19.2	14.3	-
Cure index ($T\Delta 30 = t_{35} - t_5$)	50	16.5	13	-	2.75	-	3	12.2	7.1	-

NOTE: Serrated, large rotor used.

TABLE VI (cont'd)

CURE CHARACTERISTICS AS DETERMINED WITH
MOONEY VISCOMETER (LARGE ROTOR)

Ingredient Parts by Weight	Compound Number										
	16	19	20	21	24	32	35	38	41	50	51
Nitroso, Thiokol 5702	100	100	100	100	100	100	100	100	100	100	100
Triethylenetetramine	3			1	3	2.5	2.5	2.5	2.5	2	3
Hexamethylenediamine carbamate	1	2	2		1	1	1	1	1	2	1.25
1,4 Cyclohexane bis (methylamine)	[illegible]	1		1							
ZnO	3	3	3	3		3	3	3	3		3
MT black		8	8		4		10				
HAF black		17	17						10		
Ethylenediamine carbamate				1							
Nitroso-treated HiSil 233				12.5							
Silicone-treated HiSil					15	20	10	10	10		
EPC black								10			
HiSil 303										20	15
Temp. of test (°F)	250	200	200	200	200	210	210	210	210	212	212
Minimum viscosity	7	66	10	48	66	53	40	54	50	55	50
Scorch time (t_5 min)	11.2	10	-	-	2	5	3	1.5	1.75	-	4.75
Time of cure (t_{35} min)	16	-	-	-	11	8	9	4.2	4.25	-	-
Cure index ($t\Delta 30 = t_{35} - t_5$)	4.8	-	-	-	9	3	6	2.7	2.50	-	-

TABLE VII

DIFFERENTIAL THERMAL ANALYSIS RESULTS

Sample Identification	Tg°C	Tc°C	Td°C
Thiokol 5702	-50	-	220
Thiokol 5702 with TETA	-49	60	200
Thiokol 5702 with Diak #1	-	-	155
Thiokol 5702 with H_2N  NH_2	-	-	168
Thiokol 5702 - TETA and UROTROPIN	-	-	137
Thiokol 5702 - dicumylperoxide	-	-	133
Thiokol 5702, TETA, Diak #1, DPG, CAB-O-Sil	-	-	138

Tg = Second order transition temperature
Tc = Onset of crosslinking temperature
Td = Onset of degradation temperature

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13. ABSTRACT

Nitroso rubber, a copolymer of trifluoronitrosomethane and tetrafluoroethylene, has been made in pilot-plant quantity by the Thiokol Chemical Corporation under Army Contract DA19-129-AMC-69. Research compounding studies have been made on this pilot-plant production rubber. A variety of cross-linking (vulcanization) agents were investigated, the most effective of which was triethylenetetramine. Of a series of reinforcing fillers used, the best physical properties were obtained with HiSil 303. However, a silicone-treated HiSil 233 gave comparable results.

The curing characteristics of the nitroso rubber made in the pilot plant at the Thiokol Chemical Corporation were different from those of the material prepared in the laboratory of the Minnesota Mining and Manufacturing Company. Based on the research compounding studies made, there is no indication that rubber compounds having excellent physical properties can be made from the Thiokol Chemical Corporation nitroso rubber.

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Compounding	8					
Vulcanization	8					
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